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ARPA-NBS Program of Research On High Temperature Materials

**U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS**

NATIONAL BUREAU OF STANDARDS

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NBS Technical Notes are designed to supplement the Bureau's regular publications program. They provide a means for making available scientific data that are of transient or limited interest. Technical Notes may be listed or referred to in the open literature.

CONTENTS

	Page
1. Introduction - - - - -	1
2. Objectives - - - - -	2
3. Project Summaries - - - - -	6
3.1. Optical Constants of Titanium in the Visible Region of the Spectrum A. J. Melmed and J. J. Carroll - - - - -	6
3.2. Diffusion in Refractory Materials A. L. Drago - - - - -	6
3.3. Crystal Growth from Vapor H. S. Parker and C. A. Harding - - - - -	7
3.4. High Temperature Creep in Metals A. A. O. Rukwied, W. A. Willard and D. E. Harne - - - - -	9
3.5. Deformation and Fracture of Ionic Crystals S. M. Wiederhorn and L. H. Bolz - - - - -	14
3.6. Electronic Structure of Refractory Hard Metals J. R. Cuthill, A. J. McAlister, L. H. Bennett and I. D. Weisman - - - - -	15
3.7. High Temperature Thermodynamics E. D. West and S. Ishihara - - - - -	18
3.8. The Volatilization and Decomposition of Materials G. Mattamal, S. Straus, L. A. Dunlap and L. A. Wall - - - - -	22
3.9. Relaxations Near the Melting Point J. P. Colson and R. K. Eby - - - - -	25

ARPA-NBS PROGRAM OF
RESEARCH ON HIGH TEMPERATURE MATERIALS

Work Performed at the National Bureau of Standards
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Edited by

A. D. Franklin

Brief reviews are given of work performed during the period July 1 to December 31, 1968, on a number of projects concerning High Temperature Materials. Topics include the optical constants of titanium, diffusion of oxygen in oxides, growth of Al_2O_3 bicrystals by chemical vapor deposition, high temperature creep in copper, fracture in glass, the electronic structure of transition metal borides and related compounds, the enthalpy of pyrolytic graphite at high temperatures, the mechanism of volatilization of polymers, and the interaction between mechanical relaxation and annealing in polymers.

Key words: high temperature materials, materials properties, mass transport, diffusion, oxides, crystal growth, creep, copper, fracture, glass band structure, transition metal borides, enthalpy, pyrolytic graphite, evaporation, mechanical relaxation and polymers.

1. INTRODUCTION

The National Bureau of Standards, with support from the Advanced Research Projects Agency of the Department of Defense, is carrying out a program of research on High Temperature Materials. In this program are included projects on the properties of high temperature materials, projects on their preparation, and others designed to explain the basic phenomena limiting the use of materials at high temperatures. A summary of the results achieved in the period is given here.

2. OBJECTIVES

The failure at elevated temperatures of materials whose properties are quite adequate at lower temperatures is one of the important factors limiting the advance of Defense technology. This fact has long been recognized and the Department of Defense has in the past supported and is now supporting a great deal of research to improve the higher-temperature performance of a wide variety of materials.

The continuing success of these efforts will depend upon timely advances of the science involved in materials preparation, characterization, and property control. It is the overall objective of this program to identify some of the more important of such key problems and to try to provide the needed advances where NBS possesses the appropriate competence. Efforts will be directed mainly toward providing techniques for the preparation and characterization of research materials, providing techniques for the measurement of properties, obtaining reliable values of key pieces of data, and improving the understanding of material behavior particularly relevant to the higher-temperature performance.

In pursuit of this program, a continuing attempt is being made to analyze technological problems in higher-temperature materials use and distill from this analysis major scientific problems whose solution would advance the technology and to which NBS has the competence to address itself. A committee has been formed to steer the work on this analysis and to guide the program. While the analysis is not yet at the stage of formal reporting, certain key scientific problems of this kind have been identified and are being worked upon. Examples are given below.

The corrosion, particularly by oxidation, of structural metals at high temperatures is an outstanding technological problem. Protection by some sort of coating, either deliberately applied or formed as a passivating layer during the initial stages of corrosion, is one well-explored approach to solving the problem.

In the study of the initial phases of corrosion, ellipsometry is proving to be very important. To apply it adequately, however, the optical constants of the base metal are needed. A project is described below (Optical Constants of Titanium in the Visible Region of the Spectrum, A. J. Melmed) aimed at obtaining these data for an important structural material.

In the case of oxidation, the coating must provide a barrier to the passage of ions through the coating. Thus diffusion in oxides is an important process, and this is true also for a number of other technological problems, including sintering of ceramics, swelling of reactor fuels, and operation of ceramic fuel cells. At present the diffusion of ions in oxides is not at all well understood nor subject to anything but very crude control. Among the major reasons are lack of sufficiently pure crystals upon which to make measurements; the lack of reliable techniques for measuring diffusion rates, and therefore of reliable diffusion data, particularly for oxygen diffusion; and the lack of well-characterized grain boundaries to assess the importance of intergranular diffusion in polycrystalline ceramics and oxide films.

Several projects in this Program are concerned with studies related to these areas. Continued assistance is being given to ARPA in the scientific surveillance of a group of projects supported in other institutions by ARPA and aimed at producing oxide crystals worthy of diffusion research and at developing techniques and reliable data on diffusion in oxides. At NBS, in this present program, efforts are being made at developing a technique for the reliable determination of oxygen diffusion coefficients (Diffusion in Refractory Materials, A. L. Dragoo) and for the growing of oxide crystals, particularly with controlled grain boundaries, by chemical vapor deposition (Crystal Growth from Vapor, H. S. Parker and C. A. Harding).

Several other mechanisms operate to degrade the mechanical properties of structural metals and non-metals at higher temperatures. Creep becomes a problem in all metals at sufficiently high temperatures, but the mechanisms involved are not well understood, particularly the roles played by dislocation motion and self-diffusion for the very slowest deformation processes. A project in this field (High Temperature Creep in Metals, A. A. O. Rukwied) is concentrating on copper as a model material at the present time.

Ceramics would have great potential as structural high temperature materials but for their tendency to brittle fracture. Crack initiation and propagation processes are important. There is often chemical reaction with the environment involved, leading to stress-corrosion. Techniques are needed for studying these processes in a detailed way, to enable the role of plastic deformation, crystal orientation, surface energy, chemical reactions, and other contributing processes and parameters to be spelled out. The

Project in this area (Deformation and Fracture of Ionic Crystals, S. M. Wiederhorn) has developed a very refined technique of this kind for the study of crack propagation and is using it for fracture studies in inorganic glasses, oxides such as Al_2O_3 , and will perhaps include grain boundary fracture in Al_2O_3 if bicrystals become available.

A class of high-temperature materials that have very useful properties but that have not received as much attention as, e.g., the refractory metals and the oxides, is that of the electron-rich intermetallic compounds, such as the transition-metal carbides. The electron states are very important in determining the physical as well as electronic properties of these materials. These electron states are summarized in the electronic band structure, and progress has been made in both the theoretical calculation and experimental probing of such band structures. A project (Electronic Structure of Refractory Hard Metals, J. R. Cuthill, A. J. McAlister, L. H. Bennett and I. D. Weisman) in this area is capitalizing upon the very high-resolution NBS soft-x-ray emission facility combined with related measurements (nuclear magnetic resonance and Mössbauer spectrometry) to elucidate the band structures of transition metal borides and beryllides.

Graphite represents another class of high-temperature materials. There had been a report (from proprietary sources) that pyrolytic graphite exhibits high-temperature thermodynamic properties quite different from those of well-crystallized graphite. If true, the comparison could help shed light on the structure and properties of pyrolytic graphite. In a Project just completed (High Temperature Thermodynamics, E. D. West and S. Ishihara) a unique NBS high temperature calorimetry facility was used to show that no significant difference exists between the enthalpies of pyrolytic and well-crystallized graphites at high temperatures.

While polymers are not thought of as high-temperature materials in the same sense as ceramics, their use is limited to temperatures below a certain upper limit. In the neighborhood of and above this limit, there are "high-temperature problems" in polymers. One of these is the evaporation of polymers, used as coatings, sealants, elastomers, lubricants, etc., in vacuum environments. To control and improve the behavior of polymers in this respect, the process must be understood. A project in this area (The Volatilization and Decomposition of Materials, G. Mattamal, S. Straus, L. A. Dunlap, and L. A. Wall) is aimed at sepa-

rating the process of evaporation by molecular units from that of degradation of the molecules followed by evaporation of the fragments in model materials, and at studying the vaporization of plasticizer components.

By maintaining a continuing analysis of important needs in the science underlying high-temperature use of materials, and matching problems to NBS competence, it should be possible to have a dynamic, evolving program that contributes significantly to solving current and future high-temperature problems, and can also act as a source of assistance and advice to ARPA in this important field.

3. PROJECT SUMMARIES

3.1. Optical Constants of Titanium in the Visible Region of the Spectrum

A. J. Melmed and J. J. Carroll

Metallurgy Division
Institute for Materials Research

The purpose of this project is to measure optical constants (complex index of refraction) in the visible light spectrum for clean titanium surfaces in ultra-high vacuum, using the technique of ellipsometry. Since Ti is an hcp-structured metal it is expected that optical properties will vary with surface crystallographic orientation. Therefore single crystal as well as polycrystal specimens will be investigated.

Some (0001)-oriented single crystal specimens were prepared and optical constants were measured in ambient air. We are awaiting construction of a new ellipsometry-LEED tube in order to proceed with the planned ultra-high vacuum measurements. Because a prospective employee, counted upon for the major portion of this project, elected not to join the NBS staff, progress on this project has not been as rapid as planned.

3.2. Diffusion in Refractory Materials

A. L. Dragoo

Inorganic Materials Division
Institute for Materials Research

The objectives of this program are to measure and to interpret diffusion of oxygen in single crystal oxides. Experimentally these two objectives consist of (1) extending the sensitivity of present techniques so that the O^{18} distribution in a single crystal sample can be measured, (2) comparing the results of these concentration profile measurements with measurements of the rate of exchange of O^{18} between the gas phase and the oxide crystal, (3) determining the dependence of the diffusion rate on temperature, oxygen partial pressure, impurities in the solid, etc. and (4) obtaining reliable diffusion and exchange coefficients. In regard to interpretation, this objective translates into determining the mechanisms for oxide ion diffusion in the

oxide studied. Currently, rutile (TiO_2) is the oxide of interest. A mass spectrometer is being used to determine isotope ratios.

To date, the research has been concerned principally with developing an analytical technique for determining the O^{18} content of small ($\sim 1\mu\text{g}$) samples of oxide. AgCN reduction to produce CO_2 has been discarded because yields were too low. Presently, a graphite reduction chamber is being set up to liberate the oxygen as CO. This appears to be both kinetically and thermodynamically feasible above 1800°C if a platinum catalyst is used. Since CO_2 is more desirable than CO for mass spectrometric analysis, the conversion of CO to CO_2 at a hot filament is being studied. Several different metals are being tested.

The diffusion equation was solved for a problem involving competition between two different solid diffusion media for the isotope in the gas phase. This problem arose out of the need to separate the effects of the sample and of a partially O^{18} -enriched alumina furnace tube. The problem was solved assuming equilibrium with the gas at the surfaces of both materials, a continuous initial distribution in the walls of the tube and an initially normal O^{18} content throughout the solid. It appears to be possible to extend the mathematical procedure to problems of a finite rate of exchange.

3.3. Crystal Growth From Vapor

H. S. Parker and C. A. Harding

Inorganic Materials Division
Institute for Materials Research

The objectives of this program are to obtain an understanding of the parameters controlling perfection, purity, and dopant distribution in single crystals prepared by the vapor growth technique and to extend the technique to a wider range of refractory oxide materials. Specifically, it is planned to investigate the effect of process variables such as partial pressures of reactants, total pressure, and growth temperature on the growth process and to utilize this information to further develop the technique for the growth of well-characterized research materials of greater physical perfection and chemical purity. Aluminum oxide has been chosen as the first experimental material because previous

work by other investigators has shown the suitability of the vapor growth technique for Al_2O_3 and because of its technological importance and scientific interest as a research material.

During this reporting period, emphasis has been placed on the growth of Al_2O_3 bicrystals containing tilt boundaries. Results have been encouraging. Thus far, bicrystals have been grown with either 15° or 30° tilt misorientations between the c-axes in the mirror plane (i.e., rotation about an a-axis). The maximum size of bicrystal obtained thus far is of the order of $3 \times 5 \times 20$ mm during a growth period of 24 hours. Attempts to extend the growth period have been unsuccessful thus far, because of secondary nucleation on the growing faces and departure of the boundary from the desired propagation direction. The possibility of grinding and repolishing the crystal at intervals during the growth cycle to overcome the secondary nucleation is being investigated. The problem of maintaining the growth of the boundary in the desired direction, particularly in the case of the 30° misorientation, is more critical and may represent a limitation on the maximum size which can be grown, at least in this particular orientation. Preliminary optical and x-ray examination of the 30° specimens indicate that the favored propagation direction may be a (4483) plane. Microscopic examination indicates the boundaries to be of good quality, free of inclusions or porosity.

As anticipated, the perfection of the seed crystals has a marked influence on the perfection of the resulting bicrystal. The use of Verneuil boules has been abandoned in favor of Czochralski-grown ruby as a source of seed plates. No trouble has been experienced with diffusion of chromium from the seed into the growth regions. The use of vapor grown crystals for seed plates is also being investigated.

Reaction of AlCl_3 vapor with the nickel-alloy injector on the growth chamber is felt to be a possible source of contamination in the crystals as evidenced by deposits on the inner surface of the chamber. The use of an inner Al_2O_3 tube to minimize this effect is currently being evaluated but the results of chemical analysis are not yet available.

During the next reporting period, it is planned to complete the characterization of the bicrystals and to submit a paper for publication summarizing the results of the growth of bicrystals of Al_2O_3 by the vapor growth technique.

3.4. High Temperature Creep in Metals

A. A. O. Rukwied, W. A. Willard, and D. E. Harne

Metallurgy Division
Institute for Materials Research

Objectives:

The objectives of this project are to investigate the microscopic mechanisms that control high temperature creep, i.e., creep occurring at temperatures $T > T_m/2$, where T_m is the melting point. More specifically it is planned to study:

- (a) the effect of temperature on creep rate and compare this to the temperature dependence of dislocation velocity,
- (b) the effect of stress and compare this to measurements of dislocation density carried out by etch pit techniques,
- (c) study the effect of stacking fault energy through its variation in an alloy series and
- (d) study the effect of grain boundaries by comparison of studies in single crystals and polycrystals

Equipment:

A commercial Baldwin creep machine has been equipped with a high temperature furnace, using an inert gas, Helium, which is being highly purified by a system containing a cryosorb trap. This machine provides data for complete creep curves and has provision for stress and temperature cycling, the latter at relatively slow rates only.

Additional testing equipment has been designed and built in a joint effort with SATEC SYSTEMS, Inc., to conduct creep tests on single crystals at constant resolved shear stress as well as on polycrystals at constant true stress or even at constant load in high vacuum (10^{-4} - 10^{-6} N/m²) or at any intermediate pressure of inert gas between atmospheric pressure and high vacuum. The machine is capable of performing the rapid temperature and stress changes needed for activation analysis.

The control of the resolved shear stress is accomplished by the use of an analog computer, which calculates the resolved shear stress signal from the extensometer and load cell signals. This signal is compared with a set stress signal in a PAT control unit and the error signal is used to control the pneumatic load on the specimen. This setup is also capable of conducting tests at constant resolved shear strain rate or any stress rate. This equipment has been transferred to and accepted by NBS.

Experimental Work

Creep tests on polycrystalline copper

Using the Baldwin creep machine, work has been concentrated on the temperature dependence of high temperature creep in first and second stages at three stress levels ($\sigma=10.34$, 15.51 , and 20.68 M N/m², or 1500 , 2250 and 3000 psi).

First stage creep

The analysis of first stage creep confirmed the existence of a power law, relating strain, ϵ , to time, t , in the form

$$\epsilon = \epsilon_0(T) + \beta(T) t^m, \quad (1)$$

where $\epsilon_0(T)$ is the instantaneous strain upon loading at the thermodynamic temperature T , $\beta(T)$ is a constant for fixed temperature and stress, and the number m is the time exponent. Relationship (1) was found to be valid up to about 4 min at the highest temperatures and up to about 50 hr at the lowest temperatures tested.

The time exponent, m , was found to be $1/2$ for the very first portions of the creep curves at all temperatures and stresses tested. For copper, a value of $m=1/3$ has been reported in the literature. In the present measurements that exponent does not hold at the beginning of the creep curves, but is valid at some time interval subsequent to that one described by $m=1/2$.

The instantaneous strain, $\epsilon_0(T)$, measured by extrapolating the straight lines of ϵ vs. $t^{1/2}$ to $t=0$ increases very slightly with increasing deformation temperature from 525° to 610°C at a stress of 10.34 M N/m². However, from 610° to 692°C a very strong increase of $\epsilon_0(T)$ with deformation temperature was observed, suggesting that a diffusion controlled process governs the instantaneous strain on loading at the higher temperatures, i.e.

$$\epsilon_o(T) \propto f(\exp(-U_{\epsilon_o}/kT)). \quad (2)$$

However a value for the apparent activation energy cannot be derived from the measurements since the exact functional relationship between strain and temperature is not known. At the higher stresses tested, only a weak temperature dependence of $\epsilon_o(T)$ was found within the workable ranges of temperature. Preliminary tests at a stress of 6.89 M N/m² (1000 psi) ran into difficulties due to partial recrystallization occurring at the beginning of the tests.

The quantity $\beta(T)$ is strongly temperature dependent. Assuming a thermally activated process underlying first stage creep, an approach can be made starting from an Arrhenius equation for the strain rate

$$\dot{\epsilon} = \dot{\epsilon}_o \exp(-U_I/kT) \quad (3)$$

and combining it with the empirical equation (1) differentiated with respect to time

$$\dot{\epsilon} = m \beta(T) \cdot t^{m-1} \quad (1a)$$

$$= \beta(T) / 2 \cdot t^{1/2} \quad \text{for } m=1/2 \quad (1b)$$

which results in

$$\ln(\beta(T)/2 \cdot t^{1/2}) = -U_I/kT + \text{const} \quad (4)$$

Assuming that at constant stress at various temperatures at the respective end points of the range of validity of eq. (1) a homologous structure in the material is reached, the respective creep rates, $\dot{\epsilon}_{\beta E}$, can be calculated from eq. (1b) using the experimental values of $\beta(T)$ and the creep strains, $\epsilon_{\beta E}$, at the end of the β -ranges in order to derive an apparent activation energy from eq. (4). At all stresses tested, this evaluation resulted in an apparent activation energy value close to that for self diffusion.

These results are confirmed by graphing in an Arrhenius type plot the time, $t_{\beta E}$, needed to creep a sample to the strain, $\epsilon_{\beta E}$. These results give an activation energy for self diffusion at test stresses of 10.34 and 15.51 M N/m² (1500 and 2250 psi) for both time exponents, $m=1/2$ and $m=1/3$.

Second stage creep

One of the major objectives to date of the test series was to find the temperature interval in which, according to reports in the literature, the activation energy for steady

state creep rises from some lower value to that one for self diffusion. However, within the capability of the Baldwin creep machine and with reasonably long creep tests, an activation energy which is somewhat less than the one for self diffusion (about 184 kJ/mol [44 kcal/mol]) was measured from the steady state creep rates in the two lower stress series. This seems to be true also for the series tested at 20.68 M N/m² (3000 psi). The cause for this difference can be attributed to the use of different purity copper, different inert atmosphere, (slightly) different grain size, and different loading conditions.

From the time needed to creep specimens through the second stage at both stresses, 10.34 and 15.51 M N/m² (1500 and 2250 psi), the activation energy for self diffusion could be derived.

The time to creep specimens to fracture or onset of recrystallization at the higher temperatures shows an interesting break at $\sigma=15.51$ M N/m² (2250 psi). From the temperature interval between 440° and 505°C the activation energy for self diffusion was derived. Between 525° and 562°C, however, one finds an apparent activation energy which comes closer to the one for recrystallization. Since in this higher temperature interval the strain is proportional to $t^{1/3}$ until fracture, it seems that the specimens are never crept beyond stage I and that they recrystallize, once enough energy is stored in stage I deformation. At the lower temperatures the specimen fracture is not due to recrystallization but is due to a process governed by self diffusion. Comparison between the stress levels at $\sigma=10.35$ and 20.68 M N/m² (1500 and 3000 psi) shows, that the former leads to the activation energy for self diffusion, while the latter thus far seems to tend more to the value for recrystallization. This observation seems to be important with regard to the definition of stage II.

Creep of single crystalline copper

Since the SATEC testing equipment has been accepted only recently, no results are available at this time. Since crystalline test specimens of specified orientation are on order from a commercial company. Delivery of these specimens is nearly completed. Furthermore, techniques for profiling straight cylindrical single crystals into buttonhead specimens have been explored.

Study of growth structure in test material

Etch pit studies aimed at measuring the dislocation density in the polycrystalline copper under various conditions of creep, encountered difficulties in continuously cast material in that a substructure was found which probably is due to segregation of impurities during freezing of the metal from the melt. This substructure cannot be destroyed by cold working and subsequent annealing treatment unless the temperature is raised very close to the melting point ($T \approx 1050^{\circ}\text{C}$). A closer investigation of this phenomenon with help of transmission electron microscopy, electron and ion probe microanalysis is under way. The purity of the material has been re-checked by several types of analyses and found to be in the range of 99.99% to 99.999% Cu before and after testing.

There are extensive reports in the literature on cellular growth in alloys, however, a second constituent is usually present at a much higher concentration than 1 to 10 ppm. The cell walls are reported to consist of an enrichment of the second constituent, second phase particles or even massive precipitation of a second phase depending on the concentration and the solubility of the second constituent in the matrix. In addition dislocation networks have been observed which apparently are needed to accommodate the second constituent. In our metal, the electron probe could not detect any foreign atoms within that substructure. An ion probe thus far produced an integral analysis of the specimen surface which resulted in the detection of 3 ppm carbon, in good agreement with a chemical analysis (3.5 ppm C). Transmission electron microscopy however did detect dislocation arrangements, the locations of which are in agreement with the optically observed phenomenon, but so far no second phase particles could be seen.

The transmission electron microscopy work was done in collaboration with Dr. A. W. Ruff, NBS.

3.5. Deformation and Fracture of Ionic Crystals

S. M. Wiederhorn and L. H. Bolz

Inorganic Materials Division
Institute for Materials Research

The objective of this program is to study and understand the fracture processes in ceramic refractories and to determine the effect of plastic deformation and environment on fracture. Fracture mechanics techniques are being used in this study to determine fracture surface energies, parameters that give precise measurement of resistance to splitting. Experiments are being conducted on glass because of its commercial importance and because it serves as a model material for brittle failure. Research on glass has two goals: one, a determination of the inherent resistance of glass to fracture and two, a determination of environmental effects on fracture.

During the last six months, the stress-corrosion reaction of water on six glass compositions was studied. Crack velocity data was obtained in liquid water as a function of temperature and applied load. The data obtained satisfies the Charles-Hillig theory which assumes that stress-corrosion is due to a stress-enhanced chemical reaction at the crack tip. Apparent activation energies for the stress-corrosion reaction were 138 kJ/mol (33.0 kcal/mol) for fused-silica, 128 kJ/mol (30.7 kcal/mol) for boro-silicate glass, 121 kJ/mol (29.0 kcal/mol) for aluminosilicate glass, 106 kJ/mol (25.3 kcal/mol) for soda-lime glass, and 105 kJ/mol (25.2 kcal/mol) for lead-alkali glass. These data are being analyzed in hopes of establishing the particular chemical reactions controlling crack propagation in glass. It is interesting that the lowest two activation energies were obtained on glasses that contained large amounts of alkali ion, suggesting an important role for these ions in the stress-corrosion reaction.

Paper Published During Report Period

S. M. Wiederhorn, "Moisture Assisted Crack Growth in Ceramics", International Journal Fracture Mechanics 4, 171-7 (1968).

3.6. Electronic Structure of Refractory Hard Metals

J. R. Cuthill, A. J. McAlister, L. H. Bennett, I. D. Weisman

Metallurgy Division
Institute for Materials Research

The objective of this project is to study relationships in the changing density of states upon compound formation in refractory hard intermetallic binary compounds. Such information is expected to contribute to the understanding of basic relationships useful for the design of high temperature alloys with prescribed properties.

Measurements will be made which give information on aspects of the electronic structure, such as density of states and internal magnetic and electric fields as a function of temperature, important to an understanding of bonding and the physical properties of refractory hard binary intermetallic compounds. The representative class of compounds are those which exhibit metallic character and are characterized by one constituent element having an incomplete d- or f-shell and the other constituent element having no d- or f-electrons. The various series of borides, aluminides, beryllides, and silicides of the metals of the first and second transition series are compounds of the above type. Compounds to be included in the experimental program will be selected from those meeting the above criteria, on the basis of the possibility of obtaining well-characterized samples. Soft x-ray (valence band) spectra will be obtained from the compounds as a function of temperature. The emission spectrum is related to the density of occupied states in the valence band. Corroborative information on internal magnetic and electric fields and the shape of the density of states at the Fermi energy will be obtained from NMR, Mössbauer, magnetic susceptibility, and electronic specific heat measurements.

The efforts thus far have been in four categories:

(1) Preparation of transition metal-borides; (2) Properties of transition metal-borides; (3) Properties of transition metal-beryllides; (4) Properties of transition metal-aluminides. Each of these is discussed in turn.

(1) Preparation of transition metal-borides. Attempts, reported previously, to prepare specimens by various melting procedures were largely unsuccessful. However, acceptable material in powder form of the following borides, as revealed by metallography, x-ray diffraction, and character of the

NMR spectra, has been obtained from commercial sources for: TiB_2 , VB_2 , Cr_5B_3 , CrB , CrB_2 , Fe_2B , NbB , and CoB . Having examined the powders in this manner, some of the same material of those found acceptable have been returned to the producer to fabricate rod-shaped specimens for soft x-ray spectroscopy.

(2) Properties of transition metal-borides.

- (a) Preliminary soft x-ray spectra were obtained on the only samples available, the initial arc-melted VB , CrB , FeB , and Ni_3B . Good intensities in the transition metal spectra were obtained (good boron spectra were obtained and reported earlier). Useful results should be possible as soon as good specimens are obtained. Significant variations in spectral shape, width, and peak energy between the elemental and combined form of the constituent were noted.
- (b) Preliminary Knight shift determinations on both the boron and the transition metals were made on most of these borides. The boron Knight shifts in both the monoborides and diborides are of the order of 0.1% or less. The transition metal Knight shifts in these borides are significantly larger, but are still below 1%.
- (c) The hyperfine magnetic fields and quadrupole interactions in Fe_2B were studied in our laboratory and have been reported on. ^{57}Fe Mössbauer spectra and both the c.w. and pulsed NMR of the ^{11}B in the 4.2 to 373 K temperature range were determined. NMR frequency dependence upon pressure of ^{11}B in Fe_2B was recorded up to 680 M N/m² (6.8 k bar). The frequency dependence upon temperature was found to deviate from the expected $T^{3/2}$ relationship below 104 K. This deviation is explained on the basis of a spin wave gap arising from the magnetocrystalline anisotropy and Lorentz fields.

(3) Properties of transition metal-beryllides.

(provided gratis by the Brush Beryllium Company)

A number of beryllides in addition to those reported earlier, namely ZrBe_2 , MnBe_8 , FeBe_{11} , NbBe_3 , ZrBe_{13} , CrBe_2 , TiBe_{12} , VBe_{12} , WBe_2 , and WBe_{12} have been examined in our laboratory and found to be nonmagnetic and nonsuperconducting above 1.4 K.

(4) Properties of transition metal-aluminides. An improved NMR pulse echo technique was developed in our laboratory and applied to the study of the distribution of electric field gradients at the various nuclear sites in NiAl. NiAl has the cubic cesium chloride structure but although the phase is stable over a wide range of stoichiometry, there are deviations from cubic symmetry associated with departures from the equiatomic ratio. This deviation from cubic symmetry introduces a distribution of electric field gradients at the various nuclear sites. The NMR pulse-echo technique is a unique method for getting information on the distribution of these field gradients. In the present work, a variation of the technique was developed and shown theoretically and experimentally to enhance the spin echo pattern by a factor of 5 over what had been achieved previously. Preliminary data on the quadrupole distribution at the Al sites was obtained. Also, soft x-ray spectra have been obtained from NiAl which have been included in a paper published in the Journal of Applied Physics.

Papers Published in the Course of This Research

1. Resonance Studies in Ferromagnetic Fe_2B and Fe_2Zr , I. D. Weisman, L. J. Swartzendruber, and L. H. Bennett, presented at the 1968 Conference on Magnetism and Magnetic Materials, New York, N.Y., November 1968, and to be published in the Physical Review.
2. Ferromagnetism in CrBe_{12} , N. M. Wolcott and R. L. Falge, Jr., Phys. Rev. 171, 591 (1968).
3. Nuclear Magnetic Resonance Studies in Paramagnetic and Ferromagnetic CrBe_{12} , N. M. Wolcott, R. L. Falge, Jr., L. H. Bennett, and R. E. Watson, Phys. Rev. Letters 21, 546 (1968).
4. Magnetic Behavior of Intermetallic Compounds of Beryllium, N. M. Wolcott, R. L. Falge, Jr., and L. H. Bennett, presented at the 1968 Conference on Magnetism and Magnetic Materials, New York, N.Y., November, 1968, and to be published in Journal of Applied Physics.
5. Quadrupole Echoes in Solids, I. D. Weisman and L. H. Bennett, to be published in the Physical Review.
6. Soft X-Ray Spectroscopy of Alloys: TiNi and the Ni-Al System, J. R. Cuthill, A. J. McAlister, and M. L. Williams, J. Appl. Phys. 39, 2204 (1968).

3.7. High Temperature Thermodynamics

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Measurements of the enthalpy of pyrolytic graphite at high temperatures were undertaken because there are no published data for this material and the proprietary information available indicated large differences from other graphites. If these differences were real, they would be of considerable scientific interest; if not, the engineering data need to be corrected.

The experimental work has been completed. Twenty-three experiments were carried out with pyrolytic graphite and six direct comparison experiments were carried out with spectroscopic grade CCH graphite. No significant differences were observed, so all data were fit by method of least squares to obtain the equation

$$H_T - H_{298.15} = A \ln(T) + BT + CT^2/2 + D \quad \text{k J/mol} \quad (1)$$

where

$$A = -5.77540$$

$$B = 2.59895 \times 10^{-2}$$

$$C = 1.23817 \times 10^{-6}$$

$$D = 25.1021$$

$$\text{Estimated standard error} = \pm .093\%$$

The estimated standard deviation is 0.09%, where the estimated standard deviation, s , is given in terms of the differences, r , between the equation and the observed values, the number of experiments, n , and the number of constants (4) in equation (1).

$$s = \sqrt{\sum r_i^2 / (n-4)}$$

This precision sets a very small upper limit on possible differences between pyrolytic graphite and spectrographic grade graphite.

The experimental data are presented in the table. In the first column are the furnace temperature and the dates of the experiment. The chronology of the experiments was planned to be random in order to uncover any systematic error associated with aging of the furnace or other parts of the apparatus. Randomness was lost for some of the highest temperatures when some of these experiments were repeated to eliminate a small systematic error due to the insufficient furnace warm-up time before taking data.

In the second column, the enthalpy data are given, corrected for the small difference between the final calorimeter temperature and 298.15 K. In the third column the moles of graphite in the sample are given along with the designation of the type of graphite. The weights of samples decrease slightly with time, probably due to abrasion in handling.

In the fourth column, the observations are converted to enthalpy per gram atomic weight of graphite and in the fifth column are given the percent differences between the observations and eq. (1).

The graphite specimens were analyzed spectroscopically for the metals.* The largest impurity was copper, which was less than 0.01%. Iron, which was found to be less than 0.005% in an unused pyrolytic graphite sample was not detected in the sample used. Titanium, which was not found in the unused sample, was less than 0.005% in the samples used. The original impurities and the contamination in use are therefore considered negligible. The enthalpy was not corrected for impurities.

The enthalpy of pyrolytic graphite does not differ by as much as 0.1% from the enthalpy of spectroscopic grade graphite. The enthalpy calculated from eq. (1) agrees with the equation obtained in the previous measurement [1], made by somewhat less accurate techniques, to 0.1% at 1200 K and going up to 1.3% at 2500 K. Observations which report such differences should be examined carefully for the adequacy of the method.

- [1] E. D. West and S. Ishihara, Advances in Thermophysical Properties at Extreme Temperatures, "A Calorimetric Determination of the Enthalpy of Graphite from 1200 to 2600 K", ASME, 1965, pp. 146-151.

*Analyses were carried out by Mrs. M. M. Carr of the Spectrochemical Analysis Section.

Enthalpy of Pyrolytic and Spectroscopic Graphites

Furnace Temp K and Date	Heat to Calorimeter at 298.15 K	Gram-atoms C; Specimen	$H_T - H_{298.15}$ J/gaw	% Above Equation
1174.3 6-5-68	7140.8 J 2364.8 2364.8 7140.9	.305408 A* .305405 A	15638 15639	- .09 - .09
1199.6 4-1-68	2349.9 7305.9 6913.4 2350.2	 .305412 A .281312 B*	 16227 16221	 .02 - .02
1299.8 4-3-68	2676.5 7892.3 8339.5 2677.3	 .281309 B .305412 A	 18541 18540	 .12 .11
1400.1 3-28-68	3009.4 9387.3 8881.7 3007.9	 .305413 A .281316 B	 20883 20880	 .09 .07
1499.4 4-15-68	3330.3 10426.2	 .305412 A	 23234	 .03
1499.6 4-17-68	3332.5 10421.6 10432.2 3334.6	 .305412 A .305412 A	 23212 23240	 - .09 .03
1602.8 4-10-68	11530.3 3681.0 3681.8 11546.7	 .305410 A .305409 A	 25701 25752	 - .11 .09
1604.7 7-8-68	11780.8 3912.8	 .305408 A	 25762	 - .05
1699.8 3-15-68	11905.2 4009.5 4011.0 12605.6	 .281317 B .305420 A	 28067 28140	 - .15 .11

Enthalpy of Pyrolytic and Spectroscopic Graphites

Furnace Temp K and Date	Heat to Calorimeter at 298.15 K	Gram-atoms C; Specimen	$H_T - H_{298.15}$ J/gaw	% Above Equation
1700.1 3-13-68	12601.4 J 4018.3	.305420 A	28102	- .05
1899.7 3-11-68	4669.4 14779.6 14780.6 4675.6	.305416 A .305416 A	33103 33086	- .01 - .06
2001.8 4-12-68	5022.2 15912.0	.305409 A	35656	- .14
2104.4 3-21-68	5391.6 16197.0	.281322 B	38409	.17
2202.1 7-23-68	5981.9 18384.9 18384.2 5979.1	.303372 A .303372 A	40884 40891	0 .02
2202.2 7-10-68	5986.6 18485.4	.305407 A	40925	.10
2302.8 8-12-58	6332.2 19002.0 19002.1 6346.4	.290982 A .290979 A	43541 43494	.05 - .06
2398.5 7-31-68	6705.0 20659.0 20665.0 6705.7	.303338 A .303338 A	46002 46019	- .11 - .07
2494.1 8-6-68	7062.7 20311.6	.272305 B	48655	.11

*Specimen A is pyrolytic graphite; B is spectroscopic grade

3.8. The Volatilization and Decomposition of Materials

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The overall objective of this project has been to better understand the mechanism of the molecular vaporization of large organic molecules from the liquid state and particular to investigate or determine the size at which thermal decomposition is required for volatilization to occur. Our initial studies include investigation of linear alkanes and a summary of our results is shown in Table 1.

Table 1. Activation Energy for Vaporization of
Linear Alkanes, $H(CH_2)_nH$

n	MW	ϵ_v , kJ/mol		
		Sol Para Eq. (2)	Theory Eq. (1)	Exp Exp.
19	268	88	79	84
24	338	111	91	92
36	506	165	120	117
94	1318	456	229	209

Here we list the experimental activation energies, ϵ_v , for vaporization and compare them with values calculated from enthalpies of vaporization, which in turn were obtained with the equation:

$$\Delta H_v = 13.4 n^{2/3} - 0.0807 T + 12.2 \quad (1)$$

in which T is the temperature in kelvin and n the chain length.

This relationship, or more specifically the term with $n^{2/3}$, results from the theoretical model that flexible molecules vaporize as compact spheres. The numerical coefficients in the expression were determined by fitting the relation to available data in the literature. The activation energies were obtained from the enthalpies with

$$\epsilon_v = \Delta H_v - \frac{3}{2} RT = \Delta E_v - \frac{RT}{2} .$$

We also make a comparison in Table 1 with activation energies calculated from estimated solubility parameters. Since the solubility parameter,

$$\delta = \left(\frac{\Delta E_v}{V} \right)^{1/2},$$

then

$$\epsilon_v = \delta^2 V - \frac{RT}{2} . \quad (2)$$

Values calculated in this manner show an increasing deviation from the experimental value with the size of the molecules, i.e., n or MW, and indicates that the solubility parameter is not related to the actual energy of vaporization but to the maximum energy of vaporization, i.e., to the energy for the hypothetical vaporization of an extended chain. It immediately follows that the difference in the energy determined from the solubility property and that determined from the rate of vaporization is the energy for extending the chain in a vacuum.

The rates of vaporization of twenty-one commercial plasticizers were also measured. From the volatilization kinetics it was determined that eleven plasticizers were essentially pure compounds. Activation energies for vaporization for these were determined and compared to those calculated from solubility parameters (Eq. 2). These results were described in detail at the 156th National Am. Chem. Soc. Meeting, September 1968.

Since September, a series of related phenylene ether compounds have been studied. Preliminary results are shown in Table 2. These compounds are of interest as thermally stable liquids and polymers. The first group of substances listed are the meta-linked polyphenylene ethers, 3 to 7 phenylene rings. In this range the activation energy appears to be linear. The largest species (7 rings) is being purified and its activation energy for vaporization redetermined, since its listed value shows the beginning of a deviation from linearity with molecular size. The last two compounds show a 29 kJ (7 kcal) difference between the activation energy for two isomers. This is a 44% increase on going from an ortho to a para isomer, and shows the effect of molecular structure independent of molecular weight.

FUTURE PLANS

(1) Extend our vaporization studies to include the available series of glycerides (tributyrin to tristearin)

Table 2. Activation Energies for Molecular Vaporization

Compound	Formula	MW	ϵ_v experimental	
			kJ/mol	kcal/mol
m-diphenoxy benzene	$\text{H} \leftarrow \text{C}_6\text{H}_4 \text{O} \rightarrow_2 \text{C}_6\text{H}_5$	262.32	to be determined	
bis(m-phenoxy phenyl)ether	$\text{H} \leftarrow \text{C}_6\text{H}_4 \text{O} \rightarrow_3 \text{C}_6\text{H}_5$	354.41	84	20
m-bis(m-phenoxy phenoxy) benzene	$\text{H} \leftarrow \text{C}_6\text{H}_4 \text{O} \rightarrow_4 \text{C}_6\text{H}_5$	446.50	105	25
bis[m(m-phenoxy phenoxy)phenyl]ether	$\text{H} \leftarrow \text{C}_6\text{H}_4 \text{O} \rightarrow_5 \text{C}_6\text{H}_5$	538.60	121	29
m-bis[m(phenoxy phenoxy)phenoxy]benzene	$\text{H} \leftarrow \text{C}_6\text{H}_4 \text{O} \rightarrow_6 \text{C}_6\text{H}_5$	630.70	(133)	(32)
<hr/>				
4-biphenyllyl phenyl ether	$\text{C}_6\text{H}_5\text{-C}_6\text{H}_4\text{OC}_6\text{H}_5$	246.31	96	23
2-biphenyllyl phenyl ether	$\text{C}_6\text{H}_5\text{-C}_6\text{H}_4\text{OC}_6\text{H}_5$	246.31	67	16

which can be considered to be linear molecules with a branch at the centers and the series of linear sebacate esters (dibutyl sebacate to dioctadecylsebacate).

(2) Extend our vaporization studies of these compounds to include measurements of their vaporization from amorphous polymers and hence to measure vaporization controlled by the diffusion process as well as that controlled by the molecular vaporization process.

(3) If time permits to explore the effect of solubility or compatibility of the plasticizer with the polymer on the rate of vaporization.

PUBLICATIONS ON THIS PROJECT

1. "Vaporization of Linear Alkanes", by L. A. Wall, J. H. Flynn, and S. Straus, presented to Div. of Phys. Chem., 156th National ACS Meeting, Atlantic City, N.J., September 1968.
2. "Vaporization of Organic Plasticizers", by L. A. Wall, J. H. Flynn, and S. Straus, presented to Div. of Polymer Chem., 156th National ACS Meeting, Atlantic City, N.J., Polymer Preprints 9, [2], 1440 (September 1968).

3.9. Relaxations Near the Melting Point

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In the dynamic mechanical properties of mats of solution-grown crystals of polyethylene measured with a torsion pendulum, the strength of the α relaxation depends upon the temperature of annealing. Furthermore, the relaxation correlates with reciprocal thickness of the crystals suggesting that the origin of the relaxation is in the crystal surface. An important question is, "Do other structural and morphological features also change with annealing?" We have reported previously that: (1) The orientation of the crystallographic axes with respect to the edges of the crystal mat does not change, (2) the unit cell dimensions do change and the area of the basal (a,b) plane of the cell correlates with the strength of the α relaxation. However, we have now found

that another parameter of importance is solvent. Samples annealed for 4 weeks in a vacuum at 80°C, exhibit a relaxation temperature 15°C higher than that for samples annealed for 1 week in a vacuum at 23°C and then annealed for 30 minutes at atmospheric pressure at 80°C. These have identical lamella thickness, unit cell dimensions, and orientation of the crystal axes with respect to the edges of the crystal mat. We have found that this difference in temperature of the α relaxation correlates with residual solvent. The one evacuated at 23°C and annealed at 80°C contains approximately two times as much solvent as the one evacuated at 80°C. As the "23°C sample" is annealed at higher temperatures, its relaxation temperature rises to that of the "80°C sample". Possibly, this correlates with the decreasing solvent content upon annealing. We plan measurements to examine this.

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